Acta Cryst. (1964). **17**, 1604

The Crystal Structure of 2-Bromo- and 2,6-Dibromo-3,3,5,5-tetramethylcyclohexanone

By L. C. G. Goaman* and D. F. Grant

Viriamu Jones Laboratory, University College, Cardiff, Wales

(*Received* 2 *December* 1963)

2-Bromo- and 2,6-dibromo-3,3,5,5-tetramethylcyclohexanone both crystallize in the space group $P2_12_12_1$. This space group is rather unexpected in both cases but in the first case D and L enantiomorphs must separate side by side from the originally optically inactive material and in the second case internal compensation of the asymmetric carbon atoms 2 and 6 leads to a molecule with a plane of symmetry. The structures were solved by conventional heavy-atom techniques and refined by Fourier and least-squares methods. Strain in the cyclohexanone rings resulting from the short distances between axial methyl groups was found to occur mainly at C(4) where the ring angle is increased to about 120°, and at the opposite ring angle at C(1) (*i.e.* at the carbonyl group) where the angle is reduced to about 110°, the strain being very similar for both compounds. These strains are discussed and the geometry of the molecules compared with molecules based on undistorted cyclohexanone rings.

Introduction

The crystal structures of 2-bromo- and 2,6-dibromo-3,3,5,5-tetramethylcyclohexanone have been determined in order to investigate the molecular strain resulting from short distances between the axial methyl groups. Anomalous conformational equilibrium effects have been observed by Sandris & Ourisson (1958), who explained them in terms of a 'reflex' effect — a rocking apart of the axial methyl groups with a consequent deformation of the ring angles. An X-ray analysis of these structures confirms these deformations and the geometry of the strained molecules is compared with ideal cyclohexanone rings.

The structure of 2-bromo-3,3,5,5-tetramethylcyclohexanone

Crystal data

2-Bromo-3,3,5,5-tetramethylcyclohexanone (hereafter referred to as MTC) m.p. 52·5–53·5 °C.

Orthorhombic

$a = 6{\cdot}05 \pm 0{\cdot}016$, $b = 12{\cdot}24 \pm 0{\cdot}007$, $c = 15{\cdot}11 \pm 0{\cdot}009$ Å;
$D_m = 1{\cdot}34$ g.cm^{-3}, $D_x = 1{\cdot}37$ g.cm^{-3} for $Z = 4$;
$\mu = 51$ cm^{-1} for Cu $K\alpha$ radiation.

The space group was determined from oscillation and Weissenberg photographs. Absent spectra were found to be $h00$ for h odd, $0k0$ for k odd, and $00l$ for l odd, and hence the space group is determined uniquely as $P2_12_12_1$ (D_2^4). Although the bulk material is optically inactive in solution the molecule of MTC contains an asymmetric carbon atom C(2), and thus in the space group $P2_12_12_1$, any one crystal can contain either the D or the L stereoisomeride. The crystals were so poorly formed it was not possible to separate the crystals of the two stereoisomers; no chemical method of resolution has been attempted. There is, however, no doubt that the space group has been correctly determined because the three-dimensional Patterson syntheses cannot be interpreted in any other way, because an R index as low as 0·14 is unlikely to be obtained with the incorrect space group, and because of the general appearance of the final three-dimensional electron density map.

Intensity data

The crystals of MTC were of poor quality, being roughly rectangular parallelepipeds with poorly defined faces. To prevent evaporation they were coated in 'Aerolyte'. The intensities of the reflexions were recorded for the layers $h = 0 \rightarrow 5$ by the use of multiple-film equi-inclination Weissenberg photographs with Cu $K\alpha$ radiation and the intensities were measured visually. Corrections for polarization and Lorentz factors were made and approximate absorption corrections were applied by assuming a cylindrical shape. The number of reflexions recorded (725) represents only 55% of the number possible in the layers recorded. The poor crystals made it impossible to record Weissenberg photographs, about the b or c axes, of sufficient quality to give reliable intensities.

The structure determination and refinement

A three-dimensional Patterson synthesis gave the coordinates of the bromine atom and a bromine-phase three-dimensional electron-density map enabled the

---

* Present address: Laboratory of Molecular Biology, Hills Road, Cambridge, England.

Table 1. *Final coordinates and their standard deviations. MTC*

Atom	x	σ_x	y	σ_y	z	σ_z
Br	1·014 Å	0·003$_6$ Å	2·428 Å	0·004 Å	3·004 Å	0·004 Å
C(1)	1·04	0·030	4·35	0·028	0·88	0·027
C(2)	1·94	0·027	3·76	0·029	2·05	0·026
C(3)	2·44	0·026	4·95	0·030	2·90	0·030
C(4)	3·13	0·027	6·01	0·030	1·97	0·028
C(5)	2·46	0·027	6·49	0·030	0·75	0·030
C(6)	1·96	0·031	5·25	0·027	−0·05	0·027
C(7)	1·21	0·036	5·53	0·032	3·72	0·034
C(8)	3·48	0·033	4·49	0·035	3·95	0·036
C(9)	3·56	0·032	7·13	0·035	−0·14	0·034
C(10)	1·32	0·032	7·45	0·036	1·04	0·033
O	−0·06	0·016	4·05	0·020	0·73	0·021

position of the molecule to be identified and all the carbon and oxygen atoms to be located. Initially the refinement was carried out with electron-density maps and was completed by least-squares methods, an anisotropic temperature factor being given to the bromine atom and individual isotropic temperature factors to the other atoms. During this stage difficulty was experienced with the temperature factor and scaling refinements, owing to the lack of intensities recorded about another axis for correlation purposes. Alternate cycles of coordinate refinement using the complete data and temperature factor and scaling refinement for each layer separately were carried out. Four of these double cycles reduced the R index to 0·14 for the observed reflexions. A representation of the final three-dimensional electron-density map is shown in Fig. 1, the final coordinates and their standard deviations in Table 1, the bond lengths and bond angles in Table 2 and the observed and calculated structure factors in Table 3. The accuracy of the structure determination is not high but is sufficiently so to show significant strain in the molecule and this will be discussed later. The structure also shows that the bromine atom is in the equatorial position, although Sandris & Ourisson's work showed only 30% existing in solution.

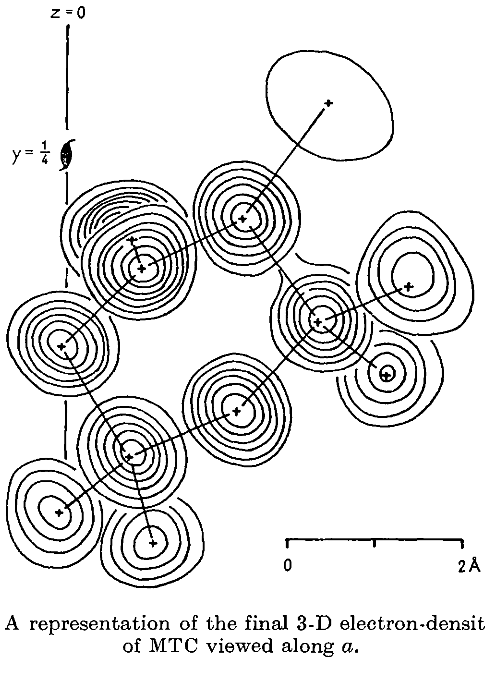

Fig. 1. A representation of the final 3-D electron-density map of MTC viewed along a.

Table 2. *Bond lengths and angles and their e.s.d.'s. MTC*

Bond	l	σ_l
C(1)–C(2)	1·59 Å	0·04 Å
C(2)–C(3)	1·55	0·04
C(3)–C(4)	1·57	0·04
C(4)–C(5)	1·47	0·04
C(5)–C(6)	1·56	0·04
C(6)–C(1)	1·59	0·04
C(3)–C(7)	1·59	0·04
C(3)–C(8)	1·55	0·04
C(5)–C(9)	1·55	0·04
C(5)–C(10)	1·52	0·04
C(1)–O	1·15	0·03$_4$
C(2)–Br	1·88	0·02$_8$

Angle	θ	σ_θ
C(6)–C(1)–C(2)	108°	2·1°
C(1)–C(2)–C(3)	107	2·1
C(2)–C(3)–C(4)	110	2·1
C(3)–C(4)–C(5)	121	2·1
C(4)–C(5)–C(6)	108	2·2
C(5)–C(6)–C(1)	110	2·0
C(2)–C(3)–C(7)	108	2·1
C(2)–C(3)–C(8)	111	2·3
C(7)–C(3)–C(8)	106	2·3
C(4)–C(3)–C(7)	113	2·2
C(4)–C(3)–C(8)	108	2·2
C(4)–C(5)–C(9)	107	2·2
C(4)–C(5)–C(10)	113	2·3
C(9)–C(5)–C(10)	112	2·4
C(6)–C(5)–C(9)	105	2·3
C(6)–C(5)–C(10)	111	2·3
C(6)–C(1)–O	129	2·3
C(2)–C(1)–O	123	2·2
C(1)–C(2)–Br	111	1·7
C(3)–C(2)–Br	116	1·7

Table 3. *Observed and calculated structure factors for MTC*

L	|Fo|	|Fc|
0 0 L		
2	32	50
4	70	76
6	14	17
8	34	38
10	13	14
12	12	17
16	10	12
0 1 L		
1	91	81
2	62	52
3	96	96
4	50	60
5	72	83
6	9	5
7	36	43
8	11	13
12	10	13
13	12	16
15	7	11
0 2 L		
0	39	45
1	6	4
2	92	80
3	9	20
5	33	28
6	11	12
7	23	20
8	38	42
9	32	36
10	20	21
11	12	13
12	12	15
0 3 L		
1	43	33
2	40	34
3	40	28
4	113	94
5	27	27
6	33	33
7	34	29
8	22	24
9	8	7
12	16	13
0 4 L		
0	17	12
1	94	84
2	46	33
3	105	90
4	11	13
5	22	18
7	23	20
8	9	9
9	33	36
11	22	22
12	9	11
0 5 L		
1	29	28
2	31	30
4	85	71
6	61	57
8	31	31
9	29	24
10	13	16
12	14	13
13	8	8
14	9	8
0 6 L		
0	43	32
1	56	56
2	11	13
3	40	36
4	23	20
5	13	13
6	10	1
7	28	29
8	24	18
9	30	26
10	9	9
11	12	16
12	9	10
13	7	6
0 7 L		
1	35	28
2	29	30
3	30	25
4	16	15
5	22	19
6	32	32
7	7	8
8	12	9
0 8 L		
0	41	42
1	25	25
2	22	20
3	11	11
4	8	12
5	17	16
6	22	21
8	16	12
10	16	15
0 9 L		
1	16	14
3	24	30
5	29	29
6	9	12
7	11	14
8	9	7
9	8	3
10	9	7
12	8	4
13	9	7
0 10 L		
0	24	28
2	21	22
5	13	10
6	10	12
8	9	11
10	11	14
0 11 L		
3	19	21
5	10	14
7	7	6
9	7	7
0 12 L		
7	9	8
0 13 L		
4	7	9
1 0 L		
1	–	19
2	94	117
4	54	62
5	32	32
6	72	79
7	29	34
8	17	23
9	23	23
10	8	5
11	11	10
14	16	16
1 1 L		
1	100	117
2	48	52
3	48	50
4	24	17
5	31	29
6	60	57
7	41	35
8	15	14
9	29	33
10	16	15
11	21	22
1 2 L		
0	33	27
1	64	59
2	100	91
3	56	46
4	63	65
5	21	23
6	27	34
7	40	41
8	17	20
9	15	15
10	20	19
11	15	14
12	12	8
13	17	15
14	9	9
1 3 L		
0	74	65
1	58	54
2	86	72
3	50	48
4	55	50
5	36	34
6	27	28
7	15	16
8	28	28
9	20	18
10	23	24
11	23	22
12	13	10
13	12	9
1 4 L		
0	36	34
1	35	27
2	55	41
3	74	66
4	23	24
5	88	78
6	16	12
7	27	27
8	16	11
9	19	18
10	17	18
11	21	20
13	12	11
1 5 L		
0	82	72
1	8	9
2	51	48
3	58	49
4	28	21
5	17	16
6	19	15
7	28	25
8	35	38
9	11	8
10	25	29
12	12	10
1 6 L		
0	25	21
1	11	15
	35	25
3	39	36
4	21	17
5	37	44
6	16	15
7	36	39
9	13	14
10	19	16
12	9	10
1 7 L		
0	31	28
1	41	40
2	29	27
3	17	13
4	15	11
5	29	28
6	25	25
7	23	20
8	23	22
9	17	14
10	13	14
12	9	14
1 8 L		
0	32	26
1	21	24
2	25	23
3	11	14
4	36	36
6	15	15
7	21	22
8	15	15
9	9	9
11	9	8
12	11	13
1 9 L		
1	33	33
2	9	9
3	16	17
4	20	20
5	13	9
6	9	8
7	16	16
8	12	9
9	13	15
11	9	13
1 10 L		
1	9	9
2	15	17
3	12	13
4	20	24
6	16	17
8	12	10
9	13	12
1 11 L		
0	12	9
1	13	16
3	16	15
2 0 L		
0	–	40
1	15	17
2	40	42
3	42	46
4	20	10
5	55	50
6	9	9
7	45	42
8	18	19
9	12	9
10	18	22
11	10	11
13	10	11
2 1 L		
0	47	67
1	15	22
2	78	73
3	54	43
4	50	32
5	48	39
6	9	7
7	35	29
8	36	37
9	20	20
10	18	21
11	17	16
12	14	16
2 2 L		
0	33	39
1	50	45
2	61	46
3	32	34
4	71	62
5	61	56
6	34	31
7	45	47
8	23	21
9	18	16
10	13	15
11	11	10
12	14	16
13	11	12
2 3 L		
0	48	51
1	53	56
2	24	19
3	49	49
4	39	34
5	33	26
6	42	46
7	23	25
8	19	17
9	26	24
10	31	33
11	21	22
12	15	14
13	9	8
2 4 L		
0	7	1
1	40	38
2	38	46
3	38	36
4	48	48
5	32	28
6	31	37
7	23	22
8	11	11
9	21	22
10	9	7
2 5 L		
1	50	56
2	32	32
3	30	28
4	11	11
5	9	6
6	30	30
7	17	18
9	21	20
10	7	5
11	14	14
12	9	8
13	13	11
14	10	9
2 6 L		
0	9	5
1	16	13
2	30	31
3	19	21
4	34	37
5	21	18
6	26	26
7	15	15
8	20	17
9	14	15
10	10	11
11	14	17
15	7	5
2 7 L		
0	31	35
1	35	39
2	10	14
3	31	26
4	10	16
5	18	13
6	21	20
7	15	13
8	23	24
9	15	14
10	11	12
11	14	15
13	12	11
2 8 L		
0	24	23
2	29	29
3	19	23
4	14	12
5	26	25
6	24	23
7	15	16
8	14	11
10	13	9
11	9	9
13	9	9
14	7	8
2 9 L		
1	12	11
2	22	24
3	21	17
5	18	14
6	12	8
8	14	16
10	14	16
11	6	7
2 10 L		
1	7	6
2	14	9
3	13	11
5	17	17
7	18	14
8	12	11
9	6	6
10	9	8
11	5	4
2 11 L		
0	16	17
2	11	11
3	7	6
4	11	11
5	9	7
6	10	7
7	7	9
8	6	6
9	7	4
10	7	10
2 12 L		
1	9	7
2	6	9
3	6	7
4	6	8
5	6	10
7	9	10
9	6	7
2 13 L		
6	6	6
3 0 L		
1	84	75
3	40	43
4	15	7
5	5	5
6	30	15
7	15	16
8	9	0
9	27	30
10	9	6
11	14	16
3 1 L		
0	11	14
1	13	14
2	20	26
3	8	6
4	47	59
5	32	32
6	36	44
7	12	5
8	31	30
9	11	10
10	10	5
11	14	9
12	13	15
13	8	5
3 2 L		
0	58	56
1	36	36
2	18	18
3	41	37
4	13	9
5	23	13
6	25	23
7	27	25
8	19	21
9	30	35
10	19	18
11	16	18
12	8	9
3 3 L		
0	8	9
1	30	28
2	23	18
3	53	52
4	33	31
5	38	39
6	30	29
7	15	16
8	19	17
9	13	13
10	8	8
12	8	6
13	12	14
14	7	5
3 4 L		
0	63	69
1	22	20
2	43	50
3	17	15
4	17	13
6	19	11
7	11	3
8	21	24
9	8	3
10	10	12
11	8	12
12	14	17
13	8	5
3 5 L		
0	6	8
1	29	23
2	19	14
3	31	39
4	10	11
5	33	35
7	29	33
8	8	3
9	10	5
10	15	11
12	8	4
13	10	11
3 6 L		
0	41	45
1	18	19
2	30	27
3	11	9
4	19	15
5	16	14
6	14	15
7	14	11
8	19	19
9	14	12
10	14	19
12	13	14
14	6	6
3 7 L		
1	29	25
2	16	16
3	20	21
4	24	25
5	23	25
6	19	17
7	18	14
8	11	8
9	14	12
10	11	9
12	8	9
3 8 L		
0	29	26
1	17	22
2	14	16
3	23	26
4	14	11
5	8	2
6	8	4
7	15	13
9	19	19
10	22	8
11	8	10
3 9 L		
0	8	6
1	13	10
2	8	8
4	23	26
5	8	8
6	17	19
7	14	10
8	10	11
9	9	8
14	3	4
3 10 L		
0	13	13
1	15	19
3	13	10
5	11	7
6	11	8
7	10	11
8	4	3
9	11	14
13	4	5
3 11 L		
0	11	6
1	10	8
2	9	15
3	8	6
4	12	14
5	7	8
6	12	14
8	8	8
9	7	7
10	5	3
3 12 L		
0	13	14
1	8	11
2	8	8
3	8	9
5	8	9
9	8	6
4 0 L		
0	–	23
1	13	14
2	37	21
3	33	31
4	13	14
5	31	31
7	45	43
8	15	12
9	6	9
10	16	15
4 1 L		
0	39	36
1	9	9
2	16	28
3	23	25
4	11	13
5	26	23
6	8	9
7	16	11
8	25	27
9	10	7
10	12	17
4 2 L		
0	20	15
1	13	12
2	25	27
3	14	16
4	20	20
5	20	27
7	9	12
8	9	4
4 3 L		
0	11	17
1	34	34
2	8	10
3	24	24
4	10	9
5	8	10
6	22	22
7	9	10
4 4 L		
0	11	6
1	19	16
2	22	20
3	8	7
4	34	37
5	11	8
6	16	15
7	11	11
8	11	13
9	12	11
4 5 L		
0	8	9
1	35	33
2	21	19
3	11	11
4	20	17
5	11	10
6	13	12
7	11	13
8	15	10
4 6 L		
0	16	11
1	25	21
2	21	20
3	7	6
4	14	17
5	15	15
6	16	17
7	10	9
11	11	6
4 7 L		
0	12	11
1	15	14
2	15	13
4	10	10
6	10	11
8	11	10
4 8 L		
3	12	17
4 9 L		
2	12	14
5 0 L		
1	28	33
2	26	25
3	15	7
4	23	28
5	31	22
6	19	24
7	24	24
8	18	12
9	15	17
14	10	15
5 1 L		
1	36	39
2	13	19
3	17	20
4	13	13
5	16	8
6	28	24
7	26	16
8	15	12
9	15	18
10	13	10
14	8	10
5 2 L		
2	16	18
3	16	17
4	24	21
5	26	21
6	28	29
7	15	17
8	13	11
9	13	14
5 3 L		
0	13	20
1	21	17
2	23	23
3	23	24
4	15	6
5	15	17
6	11	10
10	13	21
5 4 L		
0	13	10
1	21	10
2	39	36
3	19	21
4	19	14
5	28	28
6	15	10
7	15	19
8	13	11
9	15	11
10	15	13
5 5 L		
0	26	27
1	13	11
2	28	25
3	32	26
4	26	19
5	13	16
6	15	12
8	15	14
10	18	19
5 6 L		
0	24	22
1	13	10
2	15	13
3	15	18
5	26	24
7	26	25
5 7 L		
0	26	24
1	13	17
2	19	22
3	13	7
4	8	8
5	8	6
6	13	14
5 8 L		
0	21	10
1	15	16
2	15	12
3	15	12
4	13	17
5	15	13
6	13	15
5 9 L		
0	16	11
1	15	18
2	15	11
3	16	12
5 10 L		
2	13	14
6	15	13
5 11 L		
1	13	13

The structure of 2,6-dibromo-3,3,5,5-tetramethylcyclohexanone

Crystal data

2, 6 - Dibromo - 3, 3, 5, 5 - tetramethylcyclohexanone (hereafter referred to as DTC), m.p. 178–9 °C.

Orthorhombic

$a=6\cdot11\pm0\cdot025$, $b=11\cdot15\pm0\cdot004$, $c=17\cdot60\pm0\cdot006$ Å;
$D_m=1\cdot69$ g.cm^{-3}, $D_x=1\cdot73$ g.cm^{-3} for $Z=4$;
$\mu=83$ cm^{-1} for Cu $K\alpha$ radiation.

The space group was determined from oscillation and Weissenberg photographs. Absent spectra were found to be $h00$ for h odd, $0k0$ for k odd, and $00l$ for l odd and hence the space group is determined uniquely as $P2_12_12_1$ (D_2^4). The molecule of DTC contains two asymmetric carbon atoms, C(2) and C(6). It can also contain a plane of symmetry through O, C(1) and C(4) and will then be internally compensated. If this plane of symmetry is present it is rather surprising, though not impossible, that the space group is $P2_12_12_1$. There is, however, no doubt that this assignment is correct because the 3-D Patterson synthesis cannot be interpreted in any other way, because an R index as low as 0·10 is unlikely to be obtained with the incorrect space group, and finally because of the general appearance of the final 3-D electron-density map. Within the accuracy of this structure determination (see below) the molecule has been found to have this plane of symmetry.

Intensity data

The crystals of DTC were poorly formed needles of approximately circular cross-section, which bent easily and were impossible to cut without causing deforma-

Table 4. *Final coordinates and their standard deviations. DTC*

Atom	x	σ_x	y	σ_y	z	σ_z
Br	5·374 Å	0·003 Å	4·018 Å	0·003 Å	2·889 Å	0·003 Å
Br	5·579	0·004	−0·053	0·003	−1·028	0·003
C(1)	5·29	0·028	1·78	0·021	1·18	0·022
C(2)	4·50	0·025	0·66	0·021	0·45	0·021
C(3)	4·05	0·025	−0·43	0·020	1·45	0·021
C(4)	3·28	0·033	0·37	0·027	2·53	0·028
C(5)	3·96	0·027	1·46	0·023	3·29	0·022
C(6)	4·43	0·025	2·46	0·020	2·17	0·020
C(7)	5·17	0·030	−1·27	0·028	1·96	0·030
C(8)	3·10	0·039	−1·34	0·032	0·77	0·033
C(9)	2·83	0·039	2·19	0·034	4·10	0·033
C(10)	5·00	0·030	1·08	0·025	4·22	0·025
O	6·43	0·023	2·03	0·019	0·91	0·019

tion. The intensities of the reflexions were recorded for the layers $h=0 \rightarrow 5$ by multiple-film equi-inclination Weissenberg photographs with Cu $K\alpha$ radiation, and the intensities were measured visually. Corrections for polarization and Lorentz factors were made and cylindrical absorption corrections were applied. The number of reflexions observed (1074) represents 75% of the number possible in the layers recorded. It proved impossible to record suitable Weissenberg photographs for intensity measurement about the other axes.

The structure determination and refinement

The (100) Patterson projection gave the y and z coordinates of the two bromine atoms and a (100) electron-density projection using the bromine signs showed a likely position of the molecule. Three cycles of difference synthesis refinement reduced the R index from 0·30 to 0·23. A three-dimensional Patterson synthesis confirmed the bromine coordinates and a three-dimensional electron-density map based on the bromine phases was calculated. With the help of the (100) projection, and using the information that the rest of the molecule lies between two bromine atoms a certain distance apart, it was possible to find the positions of the oxygen and four of the ring carbon atoms from this first electron-density map. The

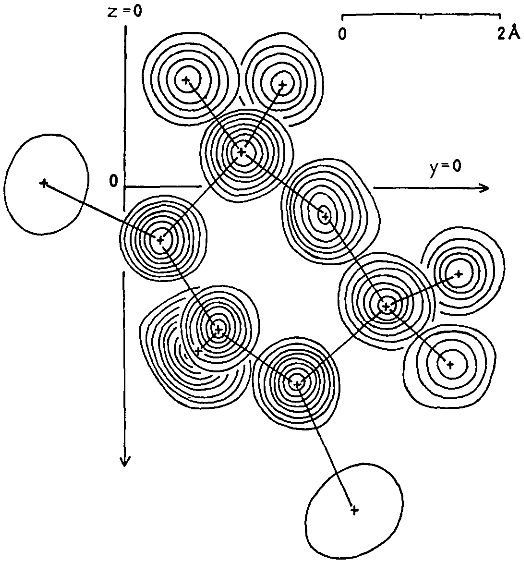

Fig. 2. A representation of the final 3-D electron-density map of DTC viewed along a.

Table 5. *Bond lengths and angles and their standard deviations. DTC*

Bond	l	σ_l
C(1)–C(2)	1·55 Å	0·032 Å
C(2)–C(3)	1·54	0·030
C(3)–C(4)	1·54	0·036
C(4)–C(5)	1·50	0·037
C(5)–C(6)	1·57	0·030
C(6)–C(1)	1·49	0·032
C(3)–C(7)	1·45	0·037
C(3)–C(8)	1·57	0·044
C(5)–C(9)	1·49	0·042
C(5)–C(10)	1·49	0·040
C(1)–O	1·19	0·036
C(2)–Br	1·96	0·021
C(6)–Br	1·96	0·022

Angle	θ	σ_θ
C(6)–C(1)–C(2)	111°	1·8°
C(1)–C(2)–C(3)	110	1·6
C(2)–C(3)–C(4)	104	1·7
C(3)–C(4)–C(5)	120	2·1
C(4)–C(5)–C(6)	104	1·8
C(5)–C(6)–C(1)	111	1·6
C(2)–C(3)–C(8)	108	1·9
C(2)–C(3)–C(7)	116	2·0
C(7)–C(3)–C(8)	108	2·1
C(4)–C(3)–C(8)	106	2·2
C(4)–C(3)–C(10)	115	2·0
C(6)–C(5)–C(9)	107	1·9
C(6)–C(5)–C(10)	112	1·9
C(9)–C(5)–C(10)	107	2·1
C(4)–C(5)–C(9)	109	2·2
C(4)–C(5)–C(10)	117	2·0
C(2)–C(1)–O	122	1·9
C(6)–C(1)–O	127	1·9
C(1)–C(2)–Br	111	1·5
C(3)–C(2)–Br	112	1·3
C(1)–C(6)–Br	108	1·5
C(5)–C(6)–Br	113	1·3

Table 6. *Observed and calculated structure factors for DTC*

L	|Fo|	|Fc|
0 0 L		
2	67	67
4	43	51
6	11	5
8	105	115
10	118	119
12	21	18
16	26	11
18	28	35
22	13	9
0 1 L		
1	70	98
2	45	55
3	110	112
4	24	17
5	54	50
6	55	47
7	24	22
8	54	57
9	54	53
10	64	64
12	54	55
13	19	20
14	25	23
15	16	15
16	25	24
17	9	11
20	27	28
22	13	11
0 2 L		
0	97	99
1	45	46
2	104	108
3	41	25
4	16	15
5	124	121
6	56	58
7	48	43
8	65	64
10	52	46
11	37	33
12	18	12
13	35	31
14	25	22
15	18	13
16	26	21
17	21	23
18	14	12
19	6	6
20	13	11
0 3 L		
1	7	3
2	26	23
3	51	55
4	171	183
5	35	34
6	106	104
7	43	37
8	45	43
9	31	28
11	24	23
12	53	46
13	24	20
14	72	71
15	15	13
16	14	10
18	20	16
22	14	18
0 4 L		
0	34	24
1	17	12
2	94	106
3	23	23
4	33	34
5	24	37
6	104	103
7	40	33
8	33	26
9	16	18
10	16	15
11	10	7
12	48	44
13	14	16
14	31	27
16	35	33
17	9	8
20	10	6
0 5 L		
1	49	51
2	16	16
3	67	64
4	82	77
5	33	26
6	62	54
8	8	8
9	67	66
10	14	15
11	23	17
12	24	18
13	19	22
14	35	32
15	13	13
16	11	10
17	16	10
19	11	10
0 6 L		
0	102	105
1	65	65
2	33	36
3	31	31
4	15	13
5	42	35
7	42	41
8	81	76
9	34	36
10	48	46
11	24	19
12	9	8
14	14	12
16	20	18
17	18	19
18	23	23
19	7	6
20	7	5
0 7 L		
2	76	82
3	11	10
6	28	24
8	43	42
9	9	5
10	29	27
12	29	29
13	9	6
16	18	17
0 8 L		
0	71	68
1	29	29
3	17	15
4	26	25
5	33	30
6	14	13
8	36	34
10	35	30
11	14	10
13	14	20
16	11	9
18	18	17
0 9 L		
1	14	7
2	20	17
3	52	44
4	42	40
5	18	17
6	27	26
7	32	24
8	6	4
9	22	21
10	18	13
11	14	16
12	24	23
13	13	11
14	6	10
15	8	8
0 10 L		
0	6	5
1	16	13
2	24	19
4	14	16
5	9	7
6	35	36
7	9	7
10	9	8
11	14	12
12	19	17
13	14	9
14	9	7
15	13	10
16	15	13
0 11 L		
1	16	14
6	17	13
4	35	33
8	19	14
14	18	17
0 12 L		
0	7	5
1	17	16
2	14	12
4	14	11
6	17	15
7	18	14
8	14	10
11	16	14
0 13 L		
1	14	12
2	14	13
3	14	13
5	8	6
6	9	8
7	13	8
10	10	9
0 14 L		
0	18	15
3	8	6
1 0 L		
1	–	10
2	116	110
3	65	61
4	109	102
5	128	129
6	48	45
8	36	31
9	14	11
12	34	31
13	49	54
14	36	39
15	26	23
17	12	13
19	6	2
22	8	12
1 1 L		
0	137	95
1	95	96
2	97	104
3	115	113
4	37	29
5	66	66
6	83	85
7	99	100
8	57	58
9	32	30
10	33	34
11	44	43
12	26	24
13	17	17
14	17	15
15	30	32
16	27	25
17	27	24
19	11	15
20	8	7
1 2 L		
0	76	70
1	65	62
2	98	93
3	138	140
4	68	64
5	59	67
6	76	79
7	73	69
8	14	10
9	40	40
10	30	26
11	49	50
12	34	37
13	39	41
14	26	27
15	26	26
16	17	15
19	16	16
20	10	13
1 3 L		
0	107	102
1	89	91
2	32	35
3	40	40
4	16	15
5	32	28
6	28	23
7	49	47
8	57	56
9	69	77
10	47	45
11	27	25
12	23	24
13	16	16
14	16	11
16	16	8
17	22	25
18	16	16
19	15	17
20	7	3
21	6	3
1 4 L		
0	5	6
1	92	91
2	97	97
3	70	75
4	14	12
5	40	36
6	42	38
7	44	47
8	35	35
9	10	10
10	41	43
11	56	60
12	32	30
13	23	20
15	16	16
16	16	17
17	12	11
19	16	14
20	12	14
21	14	14
1 5 L		
0	47	41
1	84	88
2	61	60
3	35	30
4	40	43
5	43	40
6	11	7
7	43	45
8	49	50
9	65	69
10	32	30
11	34	38
12	16	11
13	16	17
14	27	22
15	16	26
16	10	9
17	16	17
18	10	11
19	16	16
1 6 L		
0	28	26
1	30	20
2	23	19
3	49	47
4	52	56
5	61	53
6	34	36
7	38	39
8	16	13
9	15	11
10	16	10
12	26	27
13	27	28
14	15	15
15	18	17
16	7	8
1 7 L		
0	17	18
1	18	20
2	31	31
3	55	57
4	26	28
5	26	29
6	44	46
7	45	46
8	18	19
11	23	23
12	18	15
14	17	15
15	19	23
16	16	16
17	11	12
1 8 L		
0	25	27
1	18	18
2	18	18
3	32	36
4	37	43
5	47	51
6	30	31
7	12	13
8	15	13
9	17	15
10	17	16
12	10	9
13	20	24
14	14	13
15	17	18
16	8	7
1 9 L		
0	16	16
1	30	29
2	24	24
4	12	12
5	18	19
6	10	10
7	27	29
8	18	18
9	17	14
10	12	12
11	12	14
12	7	8
14	8	9
16	15	14
17	12	11
1 10 L		
0	18	24
1	19	24
2	20	24
3	17	15
4	12	10
6	17	17
7	20	20
8	10	13
9	12	13
10	17	21
11	16	19
13	8	9
1 11 L		
0	16	15
1	24	27
2	10	7
3	12	11
4	10	11
7	9	3
8	9	10
9	23	23
10	15	14
12	10	10
13	8	8
1 12 L		
0	15	11
2	16	17
3	16	14
4	11	6
5	11	7
6	15	12
8	6	6
10	7	5
11	7	8
1 13 L		
1	10	11
2	8	7
3	7	7
4	8	10
5	9	12
6	8	8
8	6	6
2 0 L		
0	–	51
1	–	116
2	39	36
3	62	53
4	18	11
5	10	6
6	18	14
7	23	18
8	29	41
9	83	95
10	49	50
11	26	17
12	17	23
13	28	32
14	25	22
17	32	33
18	27	25
19	26	23
20	18	15
21	10	8
2 1 L		
0	59	65
1	88	88
2	60	52
3	88	88
4	46	47
5	20	15
6	55	51
7	88	84
8	20	17
9	22	14
10	25	27
11	52	61
12	35	40
13	22	23
14	8	9
15	16	12
16	14	13
17	8	8
18	11	9
19	18	14
21	12	12
2 2 L		
0	21	24
1	34	38
2	93	93
3	58	51
4	94	88
5	31	22
6	20	18
7	53	52
8	60	62
9	41	41
10	36	37
11	35	33
12	22	19
13	8	7
14	21	20
15	23	24
16	20	21
17	27	23
18	12	12
19	13	13
20	8	10
2 3 L		
0	33	37
1	30	31
2	29	26
3	78	75
4	70	62
5	111	114
6	65	62
7	39	35
8	9	7
9	20	18
10	21	19
11	13	9
12	26	27
13	47	50
14	18	15
15	18	15
16	17	14
17	14	15
2 4 L		
0	33	28
1	11	12
2	34	31
3	95	94
4	18	14
5	39	39
6	22	18
7	69	72
8	30	28
9	11	7
10	25	22
11	26	26
12	6	5
13	19	20
15	32	36
16	17	15
17	17	15
20	6	5
2 5 L		
0	63	67
1	20	14
2	30	26
3	37	53
4	35	37
5	64	67
6	49	51
7	12	8
8	21	21
10	34	35
11	18	16
12	21	21
13	29	28
14	14	16
15	20	19
16	11	9
17	9	5
2 6 L		
0	49	49
1	33	36
2	58	58
3	9	8
4	31	32
5	12	11
6	10	8
7	25	21
8	28	29
9	40	50
10	36	35
11	14	12
12	10	7
13	13	11
14	13	13
16	11	11
17	18	18
18	9	9
19	10	11
2 7 L		
1	58	58
2	11	9
3	26	22
4	12	11
5	13	10
6	17	15
7	46	46
10	23	23
11	35	40
12	16	16
15	8	8
17	12	9
18	10	6
2 8 L		
0	13	11
1	44	46
2	27	24
3	13	14
4	36	39
5	8	7
6	8	5
7	8	6
8	19	19
9	31	37
10	17	14
11	15	9
12	8	8
14	16	14
17	9	9
2 9 L		
0	29	26
1	8	5
2	16	13
3	23	21
4	23	24
5	19	16
6	23	25
7	11	12
9	8	3
10	10	8
11	18	13
12	22	21
13	18	18
14	12	12
16	7	10
2 10 L		
0	10	8
1	11	10
2	21	20
3	23	24
4	22	23
5	13	14
6	11	12
7	21	21
8	17	14
10	6	5
11	9	11
13	8	5
14	9	8
15	9	11
2 11 L		
0	17	12
1	18	20
3	11	10
5	17	18
6	9	7
7	6	7
8	9	9
9	8	6
11	8	5
13	14	15
2 12 L		
0	8	9
1	9	9
2	13	15
3	11	12
4	12	12
7	17	16
8	13	12
9	6	6
10	10	8
2 13 L		
0	10	12
1	8	7
2	8	7
3	4	2
4	7	7
6	12	10
3 0 L		
1	–	52
2	–	14
4	108	118
5	36	30
6	49	66
7	22	21
8	24	22
9	25	21
10	17	14
11	17	20
12	22	24
13	19	15
14	33	45
15	6	5
16	6	6
17	17	16
18	12	13
20	3	6
3 1 L		
0	23	28
1	26	28
2	63	62
3	27	26
4	33	33
5	43	48
6	50	62
7	28	32
8	38	48
9	19	21
10	29	30
11	32	31
12	23	24
13	14	11
14	18	24
15	16	14
16	29	31
17	8	9
18	15	14
20	5	8
3 2 L		
0	59	55
1	45	43
2	40	41
3	39	40
4	52	60
5	21	21
6	64	73
7	22	23
8	29	30
9	37	35
10	27	30
11	15	11
12	25	28
13	14	12
14	21	23
15	14	14
16	9	10
17	7	6
18	12	11
19	9	9
20	9	13
21	4	4
3 3 L		
0	123	117
1	29	31
2	35	31
3	30	45
4	28	26
5	34	31
6	6	7
7	23	20
8	52	49
9	30	28
10	52	58
11	21	19
12	15	12
13	6	7
14	14	13
16	7	11
17	7	9
18	10	21
3 4 L		
0	22	18
1	43	42
2	74	80
3	38	37
4	23	22
5	9	13
6	30	24
7	19	16
8	34	34
9	12	8
10	28	31
11	23	23
12	30	33
13	12	14
14	10	11
15	6	4
16	9	13
19	7	10
3 5 L		
0	43	45
1	39	35
2	36	32
3	23	20
4	28	26
5	36	37
6	15	13
7	21	21
8	36	37
9	17	14
10	30	34
11	16	16
13	18	20
14	13	16
15	9	9
16	8	12
18	7	11
3 6 L		
0	44	41
1	7	6
2	10	16
3	29	34
4	50	56
5	14	13
6	36	37
7	24	25
8	15	16
9	16	13
10	7	6
12	28	26
13	17	17
14	25	25
15	9	8
16	8	8
17	7	6
3 7 L		
0	17	13
2	36	40
3	26	28
4	22	23
5	23	22
6	36	42
7	10	10
8	17	18
9	12	15
10	6	6
11	10	9
12	22	22
14	7	8
15	14	14
16	17	20
3 8 L		
0	6	3
1	18	19
2	15	14
3	14	13
4	25	32
5	15	16
6	10	19
7	14	14
8	15	14
9	18	22
10	8	8
11	6	8
12	11	12
13	7	8
14	18	20
15	7	9
3 9 L		
0	23	24
1	22	25
2	22	24
3	8	10
4	7	10
5	14	11
7	14	19
8	20	25
9	7	9
10	17	18
11	16	15
12	4	3
3 10 L		
0	15	17
1	7	9
2	20	27
3	6	7
4	7	7
5	7	10
6	9	13
7	9	11
8	12	12
9	7	7
10	14	16
11	7	7
12	12	14
3 11 L		
0	20	24
1	8	11
3	12	15
4	7	9
5	7	9
6	6	6
7	4	2
8	4	8
9	6	8
10	7	12
11	4	8
3 12 L		
0	7	9
1	7	6
2	7	11
3	7	13
4	4	5
5	7	9
6	7	8
7	7	11
4 0 L		
0	–	[illegible]
1	33	35
2	[illegible]	[illegible]
3	[illegible]	[illegible]
4	38	34
5	9	8
6	16	19
7	17	18
8	48	48
9	23	26
10	23	24
11	15	15
12	26	33
18	21	16
4 1 L		
1	22	27
2	43	47
3	48	50
4	11	12
5	33	37
6	19	19
7	14	19
8	31	35
9	33	36
10	21	18
11	25	23
12	16	19
13	16	28
15	11	17
4 2 L		
0	23	19
1	52	57
2	25	19
3	34	33
4	9	4
5	44	47
6	27	31
7	37	41
8	16	14
9	25	32
11	16	21
12	11	14
13	16	20
16	9	14
4 3 L		
0	13	11
1	13	11
2	16	18
3	38	33
4	44	33
5	22	28
6	18	19
7	14	18
8	22	25
11	16	7
13	22	18
14	18	23
4 4 L		
0	34	33
1	33	35
2	38	39
3	21	20
4	23	24
5	9	15
6	41	49
7	25	30
8	13	16
9	16	17
12	15	18
17	19	17
4 5 L		
0	16	13
1	38	38
2	12	7
3	37	45
4	23	22
5	22	24
7	26	31
9	26	32
11	[illegible]	[illegible]
4 6 L		
0	38	40
1	42	47
5	26	25
7	19	19
8	25	24
9	16	19
10	10	16
11	19	22
4 7 L		
1	16	16
2	23	31
3	21	23
4	16	14
8	19	20
10	21	24
4 8 L		
0	25	30
1	16	18
5	16	16
7	16	13
4 9 L		
3	19	25
5	21	22
5 0 L		
1	21	19
2	43	34
3	13	10
5	47	49
6	31	23
7	12	9
9	21	8
10	26	24
13	19	20
15	12	14
5 1 L		
0	17	20
1	32	30
2	28	26
3	39	32
4	17	16
5	17	15
6	30	21
7	45	37
8	22	20
9	21	12
10	22	18
11	29	17
13	20	17
16	7	4
5 2 L		
0	35	31
1	24	21
2	18	14
3	55	31
4	41	18
5	24	18
6	16	21
7	24	19
8	10	6
10	20	17
11	30	21
12	14	14
13	19	13
5 3 L		
0	14	8
1	40	36
2	21	20
3	26	17
4	[illegible]	14
5	14	14
6	22	17
7	13	9
8	[illegible]	13
9	31	34
10	22	16
12	21	17
13	12	14
14	10	[illegible]
5 4 L		
1	30	28
2	10	20
3	31	25
4	32	29
6	22	19
7	31	27
11	31	27
12	14	10
5 5 L		
1	34	31
2	20	19
3	13	7
4	21	20
5	17	10
6	14	20
7	23	21
8	19	12
9	21	14
10	17	14
11	13	12
14	14	13
5 6 L		
0	21	19
2	19	13
3	21	20
4	20	13
5	25	25
6	29	21
9	12	11
10	14	10
12	12	6
13	13	13
5 7 L		
0	17	13
1	14	8
2	11	6
3	29	26
5	19	12
6	17	11
7	24	25
8	17	12
10	12	9
11	11	7
5 8 L		
0	14	17
2	17	13
3	13	9
4	17	12
5	21	20
6	14	12
8	14	14
10	11	8
5 9 L		
0	13	9
1	13	12
2	14	14
4	13	10
5	12	10
8	13	15
9	12	10
5 10 L		
0	11	7
1	19	13
3	14	12

remaining atoms were found from subsequent maps and the structure was refined by least-squares methods, anisotropic temperature factors being given to both bromine atoms and individual isotropic temperature factors to the light atoms. The final *R* index was 0·10 for the observed reflexions. A representation of the final three-dimensional electron-density map is shown in Fig. 2 and the final coordinates and their standard deviations in Table 4, the bond lengths and bond angles in Table 5, and the observed and calculated structure factors in Table 6. Again the accuracy of the determination is not high, but as in the case of MTC it is sufficient to show significant strain in the molecule. The structure also shows that both bromine atoms occupy the equatorial positions.

Discussion

The arrangements of the molecules of MTC and DTC in the unit cells are shown in Figs. 3 and 4. The distances between the molecules across the screw axes parallel to *a* are short but not abnormally so — the shortest being a carbon to oxygen distance of 3·34 Å. This arrangement may cause the small asymmetry of the angles C(2)–C(1)–O (123° in MTC, 122° in DTC) and C(6)–C(1)–O (129°, 127°) (Fig. 5).

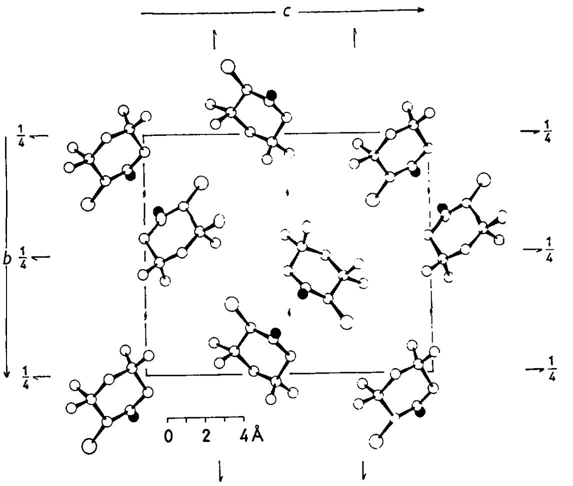

Fig. 3. The crystal structure of MTC viewed along *a*.

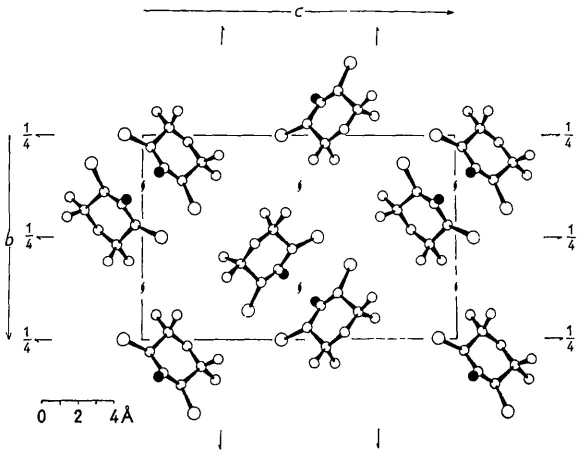

Fig. 4. The crystal structure of DTC viewed along *a*.

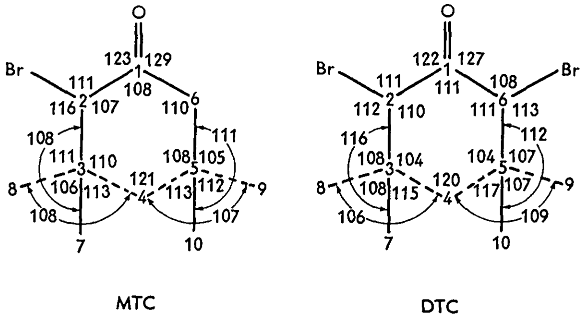

Fig. 5. The bond angles of MTC and DTC.

The bond lengths in MTC and DTC show no significant differences from the accepted values ($\sigma(l) = 0{\cdot}04$ Å, for bonds involving light atoms) and such strain introduced appears as a distortion of the bond angles. Despite an asymmetrical environment in the crystal the symmetry of the molecules (Fig. 5) suggests that the strain is introduced primarily by the bulky substituents (except for the effect on the carbonyl group mentioned above).

In both MTC and DTC the carbon atoms C(2), C(3), C(5) and C(6) lie very close (within 0·025 Å for MTC, 0·005 Å for DTC) to the best (least-squares) plane through them. It has been shown (Goaman & Grant, 1963) that if this plane is taken as a reference plane for the calculation of the atomic coordinates in the molecules, then the positions of the atoms in MTC and DTC can be compared directly with an ideal cyclohexanone ring referred to the same system of axes. This comparison shows that the six-membered rings of both MTC and DTC are distorted considerably from the ideal ring, the strain occurring mainly at the ring angles at C(1) and C(4), and moreover the strain is very similar in both MTC and DTC (Fig. 5 and Table 7).

Table 7. *The ring angles of DTC and MTC*

The standard deviation of the observed angles is about 2°

Ring angle at	Ideal cyclohexanone	DTC	MTC
C(1)	120°	111°	108°
C(2)	109·5	110	107
C(3)	109·5	104	110
C(4)	109·5	120	121
C(5)	109·5	104	108
C(6)	109·5	111	110

The angle at C(1) has been reduced from 120° to 108° (111° in DTC) and that at C(4) increased from 109·5° to 121° (120° in DTC); the steric effects have reversed the magnitudes of these ring angles. Calculation of the coordinates of the atoms in an ideal 'reversed' cyclohexanone ring, *i.e.* a ring in which the angle at C(4) is 120° and that at C(1) is 109·5°, shows that the rings of MTC and DTC resemble this ring and each other very closely. A comparison of the calculated and observed coordinates of the substituents emphasizes this resemblance (Goaman & Grant, 1963).

The cause of the ring being strained in this way is primarily the close approach of the axial methyl groups). In the unstrained ring these methyl groups would be 2·43 Å apart whereas if the angle at C(4) is increased to 120° the approach distance is increased to 3·24 Å. The calculated and observed distances between these and other substituents are given in Table 8 (Goaman & Grant, 1963). This 'rocking apart' of the axial methyl groups has been used to account for the preferential formation of the di-equatorial form of the dibromo compound (the 'reflex' effect)

Table 8. *Calculated and observed distances between substituents*

Distance between	Ideal cyclohexanone ring	Ideal 'reversed' cyclohexanone ring	DTC	MTC
Axial methyl groups	2·43 Å	3·24 Å	3·37 Å	3·30 Å
Equatorial methyl groups	4·96	4·92	4·86	4·87
Bromine–axial methyl	3·04	3·09	3·24, 3·25	3·19
Bromine–equatorial methyl	3·21	3·21	3·33, 3·36	3·35
Bromine–bromine	5·49	5·60	5·65	—
Bromine–oxygen	2·91	3·00	2·99, 2·97	2·99

(Waegell & Ourisson, 1961). In a recent paper (Waegell, Pouzet & Ourisson, 1964), the most likely conformation for these compounds has been calculated by minimizing the angular strain within the ring. It is shown that the 'rocking apart' of the axial methyl groups would result in the formation of a 'reversed' cyclohexanone ring and the values for the ring angles obtained from these calculations are shown to be in good agreement with the angles obtained by the X-ray analysis of the crystal structures described here.

Despite the large distortion in the ring angle at C(1), the atoms C(1), C(6), C(2) and O remain planar, in both compounds, within the accuracy of the determination.

Other bond angles which are significantly different from the accepted values involve the substituents and are strained to reduce the steric hindrance between the methyl groups and the bromine atoms, *e.g.* C(3)–C(2)–Br = 116° in MTC and C(3)–C(2)–Br = 113°, C(5)–C(6)–Br = 113° in DTC.

The authors' thanks are due to Prof. A. J. C. Wilson for encouragement and for the use of apparatus purchased with a Royal Society grant; to Prof. G. Ourisson, Dr C. Sandris and Dr B. Waegell of Strasbourg for suggesting the problem, providing much helpful advice, and supplying the crystals.

References

Goaman, L. C. G. & Grant, D. F. (1963). *Tetrahedron*, **19**, 1531.

Sandris, C. & Ourisson, G. (1958). *Bull. Soc. chim. Fr.* 1524.

Waegell, B & Ourisson, G. (1961). *Bull. Soc. chim. Fr.* 2443.

Waegell, B., Pouzet, P. & Ourisson, G. (1964). *Bull. Soc. chim. Fr.* In the press.

Short Communications

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 1000 *words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible. Publication will be quicker if the contributions are without illustrations.*

Acta Cryst. (1964). **17**, 1610

Crystal data (I) for some pregnenes and pregnadienes.*

By Barbara A. Haner and Dorita A. Norton, *Department of Biophysics, Roswell Park Memorial Institute, Buffalo, New York, U.S.A.*

(*Received* 30 *April* 1964)

Crystal data for seven pregnenes and pregnadienes have been determined from reciprocal lattice measurements on a General Electric XRD 5 X-ray diffraction unit, equipped with goniostat, using Cu $K\alpha$ radiation.

Space groups have been established on the basis of systematic absences and optical activity. Flotation density measurements were made and used to determine the number of molecules per unit cell. Measured and calculated densities agree within the experimental range of error (3%).

The crystal data obtained are given in Table 1.

Table 1. *Crystal data* (I) *for pregnenes and pregnadienes*

(1) 4-Pregnen-3,20-dione
(2) 5-Pregnen-3β-ol-20-one
(3) 4-Pregnen-11β,17α,21-triol-3,20-dione
(4) 1,4-Pregnadien-17α,21-diol-3,11,20-trione
(5) 5,16-Pregnadien-3β-ol-20-one acetate
(6) 5,16-Pregnadien-3β-ol-20-one
(7) 4-Pregnen-17α,21-diol-3,11,20-trione

	(1)	(2)	(3)	(4)	(5)	(6)	(7)
Formula	$C_{21}H_{30}O_2$	$C_{21}H_{32}O_2$	$C_{21}H_{30}O_5$	$C_{21}H_{26}O_5$	$C_{23}H_{32}O_3$	$C_{21}H_{30}O_2$	$C_{21}H_{28}O_5$
Mol. wt.	314·45	316·47	362·47	358·44	356·49	314·45	360·46
D_m (g.cm^{-3})	$1·15_8$	$1·15_1$	$1·27_8$	$1·33_2$	$1·13_6$	$1·10_1$	$1·30_1$
D_x (g.cm^{-3})	1·160	1·188	1·252	1·319	1·166	1·094	1·293
Z (calc.)	4	2	8	4	4	8	4
Space group	$P2_12_12_1$	$P2_1$	$P2_12_12_1$	$P2_12_12_1$	$P2_12_12_1$	$P2_12_12_1$	$P2_12_12_1$
a (Å)†	12·568	12·054	12·441	9·999	12·494	14·716	10·063
b (Å)†	13·819	11·867	30·496	23·029	27·195	26·764	23·649
c (Å)†	10·363	6·185	10·139	7·839	5·975	9·695	7·780
β	—	91·53°	—	—	—	—	—
Volume (Å^3)	1800	884	3847	1805	2030	3818	1852
Solvent	Heptane–acetone	Methanol	Ethanol	Acetone	Methanol	Acetone–methanol	Acetone

† ±0·008.

---

* This investigation was supported in part by a P.H.S. research grant (CA–6183) from the National Cancer Institute, Public Health Service.